N63 20 798

(handwritten) Pp. 201-208

(handwritten) CODE NONE

Wear and Friction of Mechanical Carbons in Liquid Oxygen as Influenced by Transfer Films

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Experimental wear and friction studies were conducted with a series of mechanical carbons sliding against metal surfaces in liquid oxygen (—298 F) at sliding velocities to 6500 feet per minute and a load of 1000 gm. The data reported shows that dense highly graphitic carbons have potential use as seal and bearing materials for liquid oxygen applications.

High density graphitic carbons with a greater oxidation resistance and a greater capability of forming a transfer film gave the lowest wear and friction. Metals that form the most stable oxide films promote greater adherence of the graphite to the mating surface. Impregnated carbons must be selected with caution because frictional heating generated during sliding can initiate hazardous reactions between oxygen and certain unstable organic compounds.

Introduction

MECHANICAL carbons are being used in seals and bearings to operate over a wide range of temperatures and environments. One area employing carbons is in liquid oxygen applications; however, the basis for selecting particular types of carbon for use in oxygen is inadequately defined. The stringent compatibility requirement imposed by the liquid oxygen environment limits the selection of impregnated carbons to be used to those of known stability in oxygen. Any unstable organic compound in the carbon could react violently with the oxygen when sufficient heat is generated during sliding. Earlier friction and wear investigations have shown that mechanical carbons can be used as self-lubricating materials for aircraft turbine engines at high temperatures of 700 F (*1, 2*) as well as in turbopump applications for rocket propulsion at liquid hydrogen temperatures, —423 F (*3*). It is feasible, therefore, that a carbon with or without adjuncts can be selected to perform satisfactorily in liquid oxygen and provide the necessary low friction and low wear required.

The low wear of mechanical carbons, as experienced in the more conventional applications, where air, moisture, or other vapors are present, is attributed to the self-lubricating characteristics of the carbon. It has been reported (*4*), that effective lubrication is accomplished only when an oriented surface layer of graphitic carbon is established on the mating surface, which in effect

results in a sliding combination of carbon on carbon rather than carbon on metal.

Reference (*1*) hypothesized that graphite lubrication depends primarily upon adherence of the graphite to the metal mating surface. This adherence can be achieved by the presence of intermediate films of adsorbed moisture, gases, or by the presence of an oxide film on one or both of the surfaces to be lubricated. The more common theory for the mechanism of graphite lubrication, which suggests the shearing of interlamellar adsorption films between the graphite platelets, does not consider affinity or adhesion of graphite to the lubricated surfaces. Earlier liquid oxygen studies (*5*) have indicated that oxide films are beneficial in minimizing wear and friction in metal–metal combinations. Therefore, since beneficial oxide films form readily on metal surfaces in liquid oxygen and graphite effectively lubricates oxidized metals (*1*), it is likely that films of graphite on the mating surface will develop readily and lubrication will be enhanced. Such film formation is essential for the lubrication to provide low wear and friction as required in dynamic seals and bearings.

The objectives of this investigation were (a) to determine the influence of graphite content on wear and friction of carbon bodies, (b) to determine the influence of adjuncts to carbon bodies on wear and friction, (c) to investigate what effect, if any, the free energy of formation of metal oxides of the mating surfaces has on wear and friction of graphite, and (d) to suggest improved seal materials for liquid oxygen turbopump applications. Carbon with varied amounts of graphite content as well as carbons modified by means of adjuncts were run in sliding contact against metals having different oxidation characteristics in liquid oxygen at —298 F. Data were obtained with a $\frac{3}{16}$-inch-radius rider speci-

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men sliding on a flat surface of a rotating 2½-inch-diameter disk. Runs were made with specimens submerged in liquid oxygen at surface speeds ranging from 1000 to 6500 feet per minute and a normal load of 1000 gm.

Apparatus and procedure

The apparatus used in this investigation is shown schematically in Fig. 1. The basic elements are a rotating disk specimen (2½-inch-diameter) and a stationary
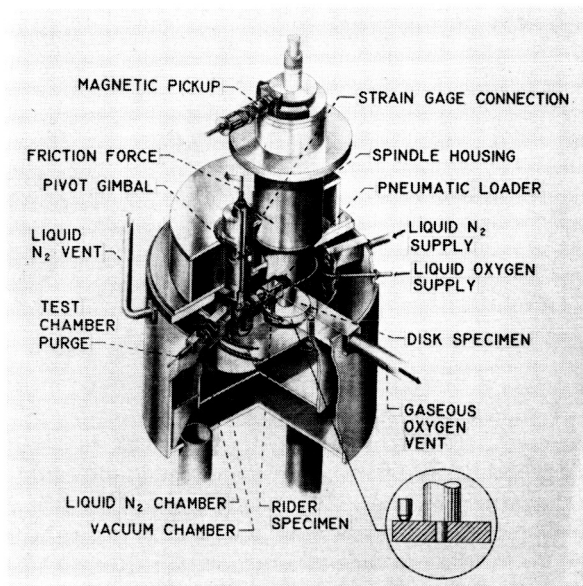

FIG. 1.   Liquid oxygen friction apparatus

hemispherically tipped rider specimen (³⁄₁₆-inch-radius) in sliding contact with the disk (see inset). The disk is rotated by a variable speed electric motor through a gear box speed increaser coupled to the specimen shaft. Disk speed is monitored by a magnetic pickup whose output is fed into a digital readout instrument.

The rider specimen is loaded against the disk by a pneumatic operated piston on the end of a gimbal mounted arm. Through this arm, pressurizing gas (dry He) is supplied. The arm is linked to a strain gage assembly for measurement of frictional force.

The apparatus consists of two sections which may be separated for changing specimens. The lower section consists of four separate chambers: The inner chamber, or test chamber, is located within a "jacket" which may be filled with liquid nitrogen for cooling. This cooling jacket is surrounded by a vacuum chamber to reduce the boil-off rate of the cooling and test fluids. The outermost chamber, or spill chamber, surrounds the three chambers and provides safe operation in the event excess amounts of the test fluid should escape. The upper section is a vacuum jacket through which passes the disk specimen drive shaft housing and the rider specimen arm assembly. A series of carbon face seals are employed to seal around the drive shaft and flexible bellows are used to seal the rider arm assembly. Fluorinated hydrocarbon oils are used to lubricate the gear box and the support bearings in the shaft housing to prevent a reaction of the oil with the liquid oxygen in case the shaft seals fail.

The test fluid, liquid oxygen in this investigation, passes through a coil within the cooling jacket before entering the test chamber near the bottom. In tests where the test fluid initially is in the gaseous state, the coil would condence the gas into the liquid state. Excess liquid and vapor are vented by a line leaving from a point near the top of the chamber. The liquid level within the test chamber is monitored by a capacitance probe and recorded on a circular chart capacitance recorder.

The cooling jacket is filled with liquid nitrogen and vented by two diametrically opposite lines. The coolant level is monitored with a carbon resistance probe, a full jacket being indicated by a sharp drop in output when the coolant absorbs the heater output.

SPECIMEN PREPARATION AND TEST PROCEDURE

The nonimpregnated carbon rider specimens were cleaned by soaking them in ACS certified acetone for 5 hours and placed in a vacuum (10^{-3} mm Hg) for 10 hours.

In cleaning the impregnated carbons it was discovered that in some cases the above procedure caused the impregnants to be leached out when they were placed in the vacuum. Therefore, the impregnated carbons were cleaned by wiping them with a soft cloth saturated with ACS certified acetone before being placed in the vacuum. All the carbon specimens were then stored in a desiccator until used.

The nonplated disk specimens used in this experiment were circumferentially finished to a surface roughness of 4–8 microinches RMS. The plated specimens, 440-C hardened to 52-54 R_c, were ground and lapped to 4–8 microinches RMS before plating (Table 1). The various platings were electro-deposited from electrolytes held in absorbent materials attached to portable electrodes (6).

All the disk specimens were then given the following cleaning treatment: (a) immersion in an ultrasonic cleaning tank filled with water, for ½ hour; (b) thorough rinsing with ACS certified acetone; (c) polishing with moist levigated alumina on a soft cloth; and (d) thorough rinsing with distilled water. The remaining water was removed using filter paper. Each disk specimen was prepared just prior to being placed in the test chamber.

After the desired liquid oxygen level was attained, the disk specimen was brought up to speed and the normal load was applied. Duration of runs was 60 minutes or less depending on the stability of operation. The frictional force was measured continuously by resistance strain gages mounted on a dynamometer ring whose output was fed into a recording potentiometer.

TABLE 1

Physical and Chemical Properties of Metal Disk Materials Used in This Study

Disk Solid	Disk Plate	Plate thick. (in.)	Standard hardness (BHN)[a]	Surface finish (μin. RMS)	$-\Delta F_{-298F}$ [b]
	Au	0.0030	110	4.2	0.30
Ag			36	6.2	7
	Ag	.0038	85	4.2	7
	Rh	.0020	925	4.0	20
Cu			82	6.2	35
	Cu	.0034	175	4.0	35
Co			101	8.4	55
	Co	.0015	—	8.4	55
Ni			68	5.5	56
	Ni	.0046	379	4.5	56
Fe			66	4.8	63
	Sn	.0042	<5	3.6	67
	Zn	.0035	50	12.0	82
Cr			120	8.4	87
	.Cr	.0042	733	4.2	87
Ti			175	7.2	110
440-C			20-54 R_c	4.0-8.0	

[a] At 75 F.

[b] Reference (*10*) (Kcal per gram atom of O_2).

Rider wear was obtained by measuring the wear scar diameter and calculating the wear volume.

Results and discussion

INFLUENCE OF GRAPHITE CONTENT ON WEAR AND FRICTION

The importance of graphite content on the wear of carbon riders is demonstrated in Fig. 2. The carbon riders were run in sliding contact with 440-C stainless steel and chromium plate in liquid oxygen at 2300 feet
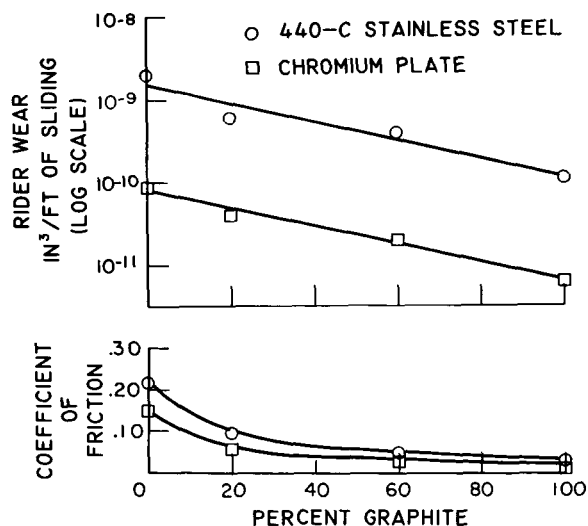

FIG. 2. Effect of graphite content on the lubricating properties of carbons in liquid oxygen (2300 fpm; 1000-gm load; duration, 1 hour).

per minute and a 1000-gm load. Rider wear decreased with increasing graphite content; the fully graphitized carbons gave the lowest wear against either 440-C stainless steel or chromium plate. In both series the wear of the amorphous carbons was about 20 times that of the fully graphitized carbon. Mechanical carbons that have operated successfully in liquid nitrogen and liquid hydrogen (*3*) have wear rates on the order of 10^{-11} inch³ per foot of sliding, which is comparable to the wear rate of the 20% graphite sliding on chromium plate (Fig. 2) in this investigation.

The low wear exhibited by the fully graphitized carbon specimens is attributed in part to their greater resistance to oxidation than those carbons of lower graphite content.

It has been reported that amorphous carbon begins to oxidize in air at 622 F and that graphitic carbon begins to oxidize in air at 842 F (*7*). It would therefore appear that, in the highly oxidative environment of this investigation, even though the temperature is at —298 F, the sliding interface temperatures were sufficient to promote a more rapid and extensive oxidation of the carbons containing little graphite and resulted in excessive corrosive wear.

It appears also that, in this investigation, the greater capability of the fully graphitized materials to form a transfer film of graphite on the metal mating surface is important in reducing rider wear. Visual inspection of the disk specimens showed that an adherent graphite film was noted best with the fully graphitized rider run on chromium plate. It was also observed that the carbons of lower graphite content did not transfer sufficient graphite to the mating surface so that the carbon was continually sliding on bare metal surfaces. As a result, the lower graphitic carbons were more susceptible to wear by abrasive processes as well as oxidation. Figure 3 is a set of photomicrographs showing rider wear scars and the transfer film of graphite on the mating chromium plate surfaces.

The distinct separation of the two curves is very significant as it points out the importance of oxides being present on the metal surfaces when graphite is to be used as a lubricant in the absence of moisture or water vapor. The selection, therefore, of mating surfaces takes on increased importance when designing dynamic seals for liquid oxygen applications. Previous friction and wear studies with graphite at elevated temperatures (*1*) suggested that the presence of beneficial oxide films promoted an affinity of the graphite for the metal mating surface and was a requirement of graphite lubrication in unconventional systems. The 20-fold reduction in wear between the two curves was the basis for a more exhaustive study as to the role played by oxide films in graphite lubrication for liquid oxygen applications. These results will be presented and discussed subsequently.

Coefficient of friction data (bottom of Fig. 2) show a downward trend as the amount of graphite is increased
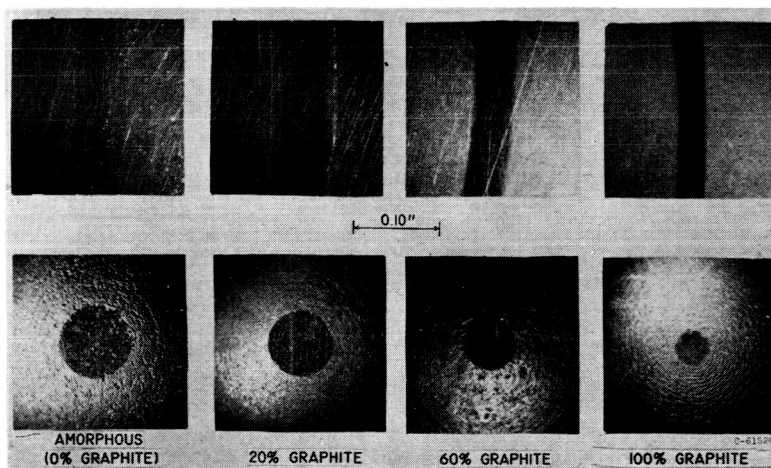
FIG. 3. Disk (top) and rider (bottom) wear areas of nonimpregnated carbons with various graphite content sliding on chromium plate in liquid oxygen (load, 1000 gm; sliding velocity, 2300 fpm; duration, 1 hour).

from 0 to 100%. The values reported are the values of friction after stabilization has occurred. The importance of graphite content in reducing friction is easily shown since the drop in friction, comparing the amorphous carbon to the 20% graphite carbon, is very pronounced: 0.22–0.10, when sliding on 440-C stainless steel and 0.10–0.06 when sliding on chromium plate. With further increases in graphite content, further reductions in the coefficient of friction are experienced with values reaching about 0.03 for the fully graphitized carbons sliding on 440-C and chromium plate.

Frictional force is the product of the effective shear area in sliding and the shear strength of the interface film (8). The harder amorphous carbons, having higher shear strength than the graphitic carbons, usually give minimum shear areas. The addition of minor amounts (20–30%) of graphite to the amorphous carbon usually gives an optimum combination of low shear strength

(from the graphite) and low shear area (from the amorphous carbon matrix) that results in minimum friction. In liquid oxygen, however, the lowest friction values were obtained with the fully graphitic carbons. It is probable that the relative influence of the cryogenic temperature on the shear area (as determined by hardness) and the chemical behavior of the oxygen on the formation of adsorbates for easy surface shear, may have altered the optimum composition.

The data of Fig. 4 are presented to show that high starting torques can be experienced with carbons of low graphite content and that for systems where high starting torques cannot be tolerated, fully graphitized carbons can provide low starting friction without extensive run-in periods. The values of friction attained after run-in are those shown in Fig. 2.

INFLUENCE OF METAL MATING HARDNESS ON GRAPHITE WEAR AND FRICTION

The differences in wear as noted in Fig. 2 between the 440-C stainless steel and the chromium plate suggested that hardness of the mating surface, usually considered important to wear of carbon type seal materials, may be responsible for the lower wear of carbons on chromium plate. A series of runs was therefore made with a hardenable stainless steel (type 440-C) of various hardnesses obtained by altering the heat treatment. In using one base metal, the variables of oxidation resistance and composition were eliminated. The data of Fig. 5 are presented to show that hardness of the mating surface from 20 to 54 Rockwell-C had no significant effect on the wear of the fully graphitized carbons when run in liquid oxygen. From this it can be deduced that graphite rider wear in liquid oxygen is influenced more by the oxidation resistance and composition of the mating surface than hardness.

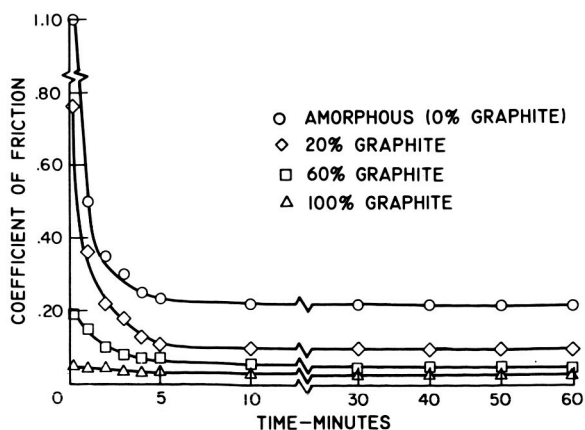Figure 5 data also show that the coefficient of friction

FIG. 4. Effect of graphite content on the friction of carbon on 440-C stainless steel in liquid oxygen (2300 fpm; 1000-gm load; duration, 1 hour).

of a fully graphitized carbon sliding of 440-C of various hardnesses remained constant within the scope of this investigation.
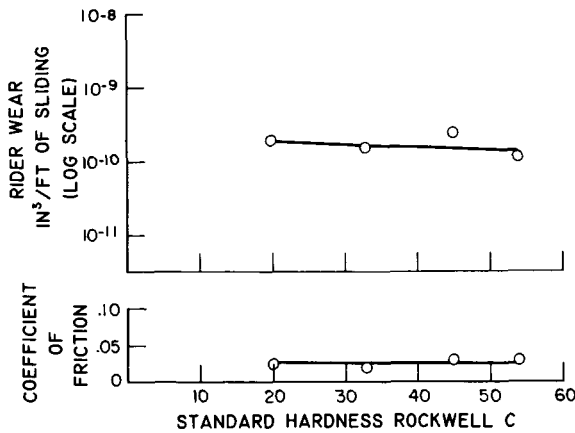

Fig. 5. Effect of surface hardness of 440-C stainless steel on the lubricating properties of graphite in liquid oxygen (2300 fpm; 1000-gm load; duration, 1 hour).

Influence of Sliding Velocity on Graphite Wear and Friction

The dissipation of frictional heat generated during sliding contact by dynamic seals in liquid oxygen could become of prime concern where substantial surface speeds are encountered. The additional heat experienced at the higher sliding velocities could increase the oxidation of the fully graphitized carbons which would lead to increased corrosive wear. A series of tests was conducted to show the effect of sliding velocity on the wear of a fully graphitized carbon rider sliding on 440-C stainless steel in liquid oxygen. Data of Fig. 6 present wear per unit distance of sliding at velocities from 1000 to 6500 feet per minute and a load of 1000 gm.
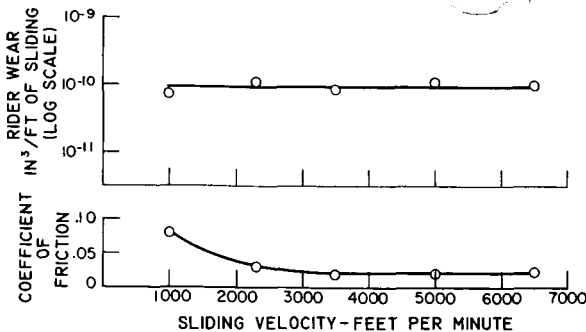

Fig. 6. Effect of sliding velocity on the lubricating properties of graphite sliding on 440-C stainless steel in liquid oxygen (1000-gm load; duration, 1 hour).

In this investigation, sliding velocity had no significant effect on graphite rider wear. Further increases in surface speeds, however, could bring about increased wear as the interface temperatures could become sufficiently

high and cause extensive oxidation of the graphite. With the addition of adjuncts to the base graphite (to increase the oxidation resistance and improve physical properties) higher surface speeds may be tolerable. The selection of adjuncts is limited, however, to those that will remain stable in the highly oxidizing environment.

The coefficient of friction (bottom of Fig. 6) shows a decreasing trend to 3000 feet per minute, after which it remains constant within the limits of this investigation. The reduction in friction between 1000 and 3000 feet per minute (0.08–0.03) is not significant as both values are well below 0.10.

The Influence of Impregnants on Wear and Friction

Two series of impregnated carbons, a 20% graphite base grade, and a 100% graphite base grade were run against 440-C stainless steel in liquid oxygen to study the effects of various impregnants on wear and friction of these carbons. The unimpregnated base grades were run against themselves for a comparison [note (A), Figs. 7 and 8].
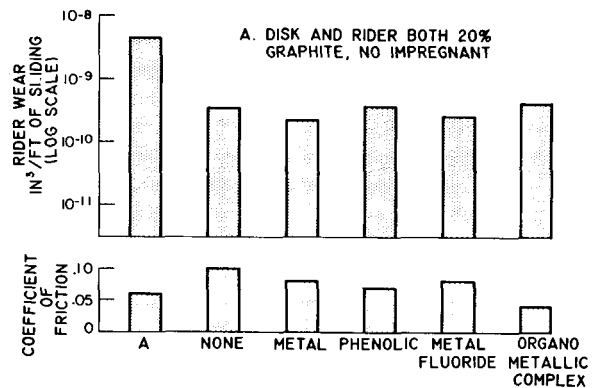

Fig. 7. Effect of impregnants on the lubricating properties of a 20% graphite sliding on 440-C stainless steel in liquid oxygen (2300 fpm; 1000-gm load; duration, 1 hour).
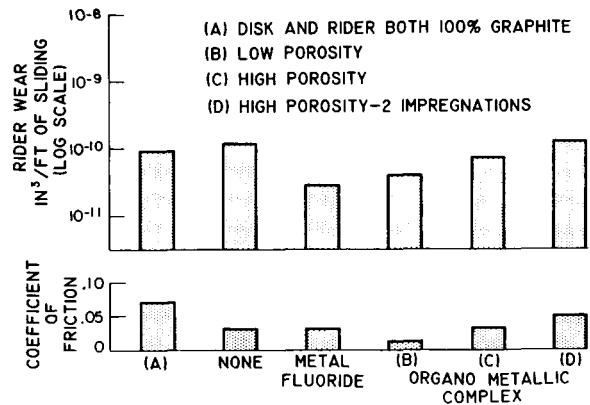

Fig. 8. Effect of impregnants on the lubricating properties of a 100% graphite sliding on 440-C stainless steel in liquid oxygen (2300 fpm; 1000-gm load; duration, 1 hour).

The wear data for the 20% graphite series, presented in Fig. 7, show that the impregnant had little effect on the wear of the carbon rider. The carbon with metal impregnation had the lowest wear: 2.2×10^{-10} inch³ per foot of sliding for this series of runs.

The coefficients of friction of the variously impregnated 20% graphite riders are presented at the bottom of Fig. 7. The addition of the impregnants had no significant effect on the frictional properties of the carbons used in this investigation. The organo-metallic complex impregnated carbon had the lowest coefficient of friction (0.04) for this series.

The data in Fig. 8 show the effect of impregnants on the wear and friction of a 100% graphite rider sliding on 440-C in liquid oxygen. In general, additive impregnants did not significantly influence wear; however, the metal fluoride impregnant and the organo-metallic complex impregnant in a low porosity carbon did reduce wear by a factor of approximately 3. The metal fluoride impregnant had the lowest wear of the two: 3×10^{-11} inch³ per foot of sliding.

This reduction in carbon rider wear indicates the oxidation resistance of the carbon was increased with the addition of the impregnant. The wear reduction is an indication of the importance of corrosive wear. The impregnant also acts to encourage a more rapid rate of graphite transfer to the metal surface which in turn reduces adhesive wear. It is important to note that the graphite riders of high porosity carbons with organo-metallic complexes (single and double impregnations) had higher wear than the low porosity graphite rider having the same impregnant.

Figure 8 also presents coefficients of friction for the same series of impregnations. The high density, organo-metallic complex, graphite rider sliding on 440-C stainless steel in liquid oxygen gave the lowest friction coefficient, 0.01.

From the results of Figs. 7 and 8, high density (low porosity) 100% graphite mechanical carbons can be considered for bearings and seals in liquid oxygen applications. The selection of impregnants should be made with caution, however, and should be limited to those having known stability with liquid oxygen.

THE INFLUENCE OF MATING SURFACE ON WEAR AND FRICTION

Friction and wear studies (9) at elevated temperatures to 700 F hypothesized that in the absence of adsorbed water or vapor, the continual presence of a proper oxide on one or both of the lubricated surfaces influenced the adherence of graphite to the mating surface and affected good graphite lubrication. A series of tests, therefore, was made to explore this hypothesis by sliding 100% unimpregnated graphite riders on various metal surfaces in liquid oxygen. These data are shown on Figs. 9 (solids) and 10 (platings).

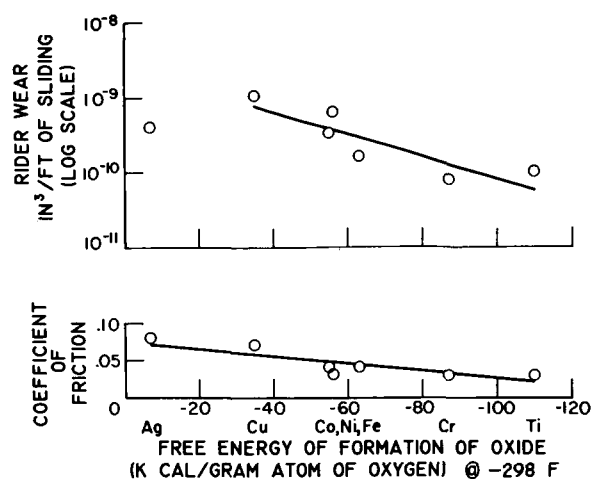The abscissa, free energy of formation of the oxide, in

FIG. 9. Effect of metal oxide formation on the lubricating properties of a 100% graphite sliding on various solid metals in liquid oxygen (2300 fpm; 1000-gm load; duration, 1 hour).
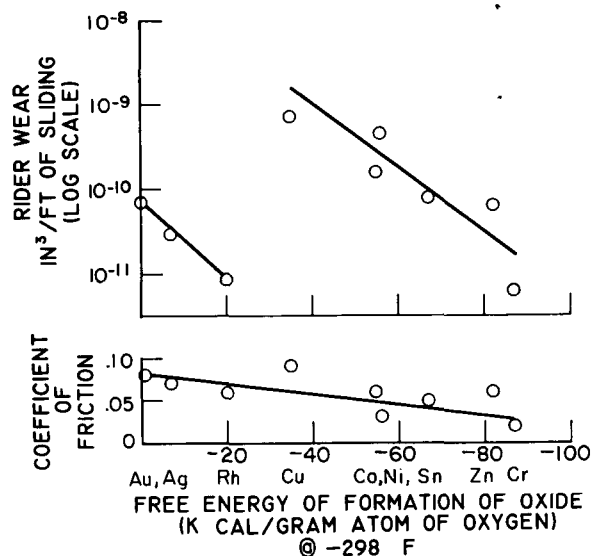

FIG. 10. Effect of metal oxide formation on the lubricating properties of 100% graphite sliding on various metal plates in liquid oxygen (2300 fpm; 1000-gm load; duration, 1 hour).

kcal per gram atom oxygen, is a measure of the ease with which the various metal oxides can form at liquid oxygen temperatures. (The more negative the value, the more stable the oxide.) The relative position of the standard free energies of formation for the oxides of the metals studied were in the same order over the entire temperature range that could be expected to result from local frictional heating on the surfaces. Reference (8) indicates that under conditions of boundary lubrication more favorable than in the present experiments, surface temperatures 900 F above the ambient might be obtained. On that basis, in these experiments, interface temperatures of about 600 F could be postulated. The surface temperatures, therefore, can be expected to range from the ambi-

ent of —298 to 600 F. The values used in plotting the relative position of the oxides formed were extrapolated from graphs down to —298 F, liquid oxygen temperature (10).

The data of Figs. 9 and 10 show a downward trend in graphite rider wear as the free energy formation of the oxide becomes more negative. This downward trend in wear is more pronounced with the plated specimens (Fig. 10) than with the solid disk specimens (Fig. 9). The break in the curve for the plated specimens, i.e., in separating the precious metals from the remaining specimens, could be caused by the presence of a contaminant, co-deposited from the electrolyte with the metal, remaining on the surface of the disk. Any contamination present in studies of this nature can have marked effect on the wear and frictional properties of the materials under investigation. The coefficient of friction also shows a downward trend with both the plated and solid disk specimens as the oxides that are formed become increasingly stable.

Those metals forming the more stable oxides developed transfer films from the graphitic carbon bodies (mechanical carbons); such films provided the most effective lubrication. The stability of the film is associated with the formation of a chemical bond between the graphite and the metal oxide. During sliding contact, penetration of the fluid (O_2) adsorbed on the surface would be achieved. Thus, with the mechanical carbon and the oxide-coated metal in intimate contact, a chemical reaction to form a carbon-oxygen-metal complex can occur. Conditions favorable for such reaction are that the contacting solids are under pressure (the yield strength of the softer slider material) and that sufficient frictional energy is available for reaction. The kinetics of such reactions are impossible to define because of transient conditions that are characteristic of sliding contact. The present reference to free energy relationships indicate the relative stabilities of potential chemical bonds in the postulated carbon-oxygen-metal complex. The chemical bonding energy is associated with stability of adhering graphitic carbon films on the mating metal surfaces as described herein. Thus, the sliding process was one of graphite sliding on a graphite film rather than graphite on nascent metal.

With those metals forming less stable oxides, it is thought that the graphite rider, through a chemical reaction, removes the bonded oxygen from the metal surface resulting in the formation of carbon monoxide or carbon dioxide. In these experiments, those metal oxides having a less negative ΔF than either carbon monoxide or carbon dioxide showed little or no graphite transferred to the mating surface. It is thought that in the liberation of these gases the nascent metal surfaces reoxidized from time to time leading to a corrosive type wear mechanism for the graphite rider. It appears that as the metal oxides become less stable, the degree of corrosive and adhesive wear increases.

Summary of results

An experimental wear and friction investigation of mechanical carbons for seals and bearings was conducted in an environment of liquid oxygen. The following results were obtained:

(1) Highly graphitic mechanical carbons had better wear and friction properties in liquid oxygen than more commonly used materials which have low graphite content. Of the graphitic materials tested, performance was best with those having high density (low porosity). Low wear values for graphitic carbon are attributed in part to its greater oxidation resistance in comparison with amorphous carbon. Also important to both wear and friction is the greater capability of the graphitic materials to form a transfer film of carbon on the metal mating surface; optimum performance appears to occur when a continuous transfer film is present on the mating metal so that the solid carbon can slide on a carbon film.

(2) Chromium plate was a better mating surface than any of the other metal platings, pure metals, or 440-C. Observations in these and other carbon wear studies suggest that the metal oxide films are of primary importance in effecting graphite lubrication. Metals forming the most stable and dense oxide films appear to perform best. The presence of these metal oxides on the mating surface promotes adherence of a beneficial transfer film of graphite from the mechanical carbons in sliding contact.

(3) Additive impregnants to mechanical carbons did not have a significant effect on wear and friction. Erratic data was experienced with impregnated carbons as compared to data for the nontreated base grade carbons; this was attributed to treatment inconsistencies. Where the use of treated carbons is preferred they should be impregnated in very nearly the final form so that the most effectively treated material will not be removed by machine finishing.

(4) Hardness of the mating 440-C material was varied from 20 to 54 Rockwell-C without significant influences on wear and friction of fully graphitized carbon rider specimens.

(5) Sliding velocity from 1000 to 6500 feet per minute had no significant effect on rate of wear or friction of fully graphitized carbon rider specimens when run on 440-C.

(6) Carbon wear problems are less severe in these liquid oxygen experiments than in liquid hydrogen studies made previously. A potential reaction hazard exists, however, with carbons in sliding contact at high surface speeds in liquid oxygen. Dense, highly graphitic materials have the greatest resistance to initiating such an oxidation reaction; any impregnants used to improve the performance of mechanical carbons in liquid oxygen applications must, therefore, have known stability in oxygen.

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